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Preparation and Physical Properties

of Doped CdBa2-x SrxCa2Cu3O8+δ Compound

Anaam W.Watana, Suad H.Aleabia, Ridha H. Risana, Kareem A. Jasima, Auday H. Shaban

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a *Department of Physic, College of Education for pure sciences Ibn -Al-Haithem, University of Baghdad, Baghdad, Iraq.*

### Abstract

In the present paper, two-steps of solid state reaction method have used to prepare the homogeneous compound CdBa2-x SrxCa2Cu3O8+δ by substitution of strontium (Sr+2) into the barium (Br+2) sites, this substitutions were taken as x = 0.0, 0.20 and 0.30. The optimum calcinations were 800 C0 and for sintering within 845-850 C0.The structural was studied by using X-ray powder diffraction (XRD) and scanning electron microscope (SEM).The XRD analysis showed the structures a polycrystalline with tetragonal diagram and the change of the strontium concentrations produce a change in lattice parameters(a, b and c-axis). c/a, density of mass (ρm ), volume fraction (Vphase) and shore hardness for all our samples .It has been found that the optimum Tc(offset) = 113 K has been found for the composition CdBa2-x SrxCa2Cu3O8+δ with x = 0.30.The A.C electrical conductivity as a function of frequency is used to find the dielectric constant. It was found that changing in dielectric constant; dielectric loss factor and tangent loss were produced by increasing the Sr concentration.

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*Keywords:* Scanning Electron Microscope, X-ray Diffraction, A.C electrical conductivity, lattice parameters, Mass density,Volume fraction and Shore hardness

1. **Introduction**

The preparation of single phase (1223) high superconductor materials is widely proven to be difficult, because of, the low phase(1212) superconductor has more preferable thermodynamically than the high phase [1].The symmetric phases are overwhelmingly intertwined to each other, in their chemical analysis [2].

In other words, preparing any of these phases as monophase is difficult particularly, especially for the phase 1223. Now, the partially replaced Tl+2 in oxygen deficient Tl-Oδ layer via positive ions have a higher oxidation states such as Bi+3, Pb+4, Cu+3 and Sb+3, it improves the stability and synthesizes material of single phase that is easily practical[ 3-6]. More concentration has

\* Corresponding author. Tel.: 009647903204147

*E-mail address:* *auday.h.s@ihcoedu.uobaghdad.edu.iq*

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been given to the synthesis of (Tl,Sb)-based because of Sb ability of reducing the melting temperature. Superconductive phase stabilizing may be increased the transition temperature of superconducting and in the structure of the crystal can be introduced more oxygen.

The replacement of (Ba) by (Sr)and that (Tl) by antimony (Sb) has the ability to improve the doping of CuO2 plane and flux pining at high magnetic fields. The critical inter-grain of current density can be improved by Sb small addition [7].It has been considered that more metalliclayer will rise at blocking layer between CuO2 conducting phase and chemical substitution on the Tl [8, 9]. Barium to the smaller Sr cation substitution in TlBa2Ca2Cu3O9-δblock of charging reservoir leads to distance decreasing of the distance between the Cu-O2 blocks of superconducting [10], which enhances the strength of interlayer coupling and causes the irreversibility line shift to be higher in the fields.

In addition, via a simple technique of encapsulation, the Sr to Tl (Sb)-1223 compound substitution can be synthesized easily, without dry box using and possessing perfect chemical stability. The studies have been presented by a new path of Cd -1223 phase synthesis, and by strontium to barium substitution with a low pressure of O2, that exploiting a high control of the achieved reaction and formation of pure CdBa2-x SrxCa2Cu3O8 δ compound permission. This compound substitutions are considered when x

= 0.0, 0.20 and 0.30.

## Experimental

By using the method of sold state reaction, the synthesis of CdBa2-x SrxCa2Cu3O8 δ (x= 0.0, 0.20 and 0.30) phases have been prepared through using pure powders materials appropriate weights of Cd2O3, BaCO3,SrCO3, CaCO3 and CuO.It considered as starting materials, according to the following chemical formula:

Cd2O3+ (2-x) BaCO3 + x SrCO3 + 2 CaCO3 + 3CuO→ CdBa2-x SrxCa2Cu3O8 δ

The samples syntheses have been carried out by two step precursor. Firstly, by using the agate mortar, the powders are mixed together (BaCO3,SrNo3, CaCO3 and CuO); a quantity of 2- propane which is sufficient for mixture homogenizing and for slurry forming during the grinding process in about (40-50) minute. Through using an oven at (2000 C), the mixture has been dried. In programmable controller furnace, the mixture has been putted to calcinations; this treatment of heat is used to remove the gas of CO2 from the mixture. To achieve this process, the powder has been heated for three hours at (800 0C) temperature with (100 0C

/hr) rate, then by similar rate the powder has been cooled at the room temperature.

Secondly, Ba2-xSrxCa2Cu3O7 precursor is mixed with Cd2O3 to obtain the nominal compositions ofCdBa2-x SrxCa2Cu3O8+δ(x= 0.0, 0.20 and 0.30). In pellets shaped like disc (1.3 cm) diameter and (0.2-0.3 cm) thick, the powder has been pressed under a pressure of (9 ton/cm2) using hydraulic press. In air at (845-850) 0C the pellets have been presenter for (20 hours) and with (200 0C/hr) rate, then at the room temperature have been cooled.

The ρ –T (resistivity vs. temperature) characteristics of these samples were measured by means of a standard d.c four-probe technique to investigate their superconducting state.

The prepared sample structure is obtained by using x-ray diffractometer (XRD). The lattice parameter, c/a, mass density and of any phase V2223ph volume fraction have been characterized everywhere [11]. The lattice a and c parameters are calculated by using a computer program based on the method of Cohen’s least square [12].

By means of �.c standard technique, the conductivity �a.c– frequency F(Hz) samples characteristics have been measured to explore their dielectric constant.

The measurements of shore hardness of produced samples are performed in air at room temperature by using the micro hardness digital tester (Durometer Shor D). The shore hardness values with three readings average are determined at different specimen surfaces locations for CdBa2-x SrxCa2Cu3O8 δ doped with x= 0.0, 0.20 and 0.30.

## Results and discussion

The series spectra of X-Ray Diffraction of CdBa2-x SrxCa2Cu3O8 δ samples with x= 0.0, 0.20 and 0.30 which is explained in Figure 1. From various samples, the XRD data is collected which is showed that all samples with polycrystalline are tetragonal structure.

Fig. 1. X-ray diffraction pattern of CdBa2-x SrxCa2Cu3O8 δ with x varying from 0 to 0.3

2500

2000

\* 1223

# 1212

1500

#

#

#

X=0

1000

X =0.2

500

0

X=0.3

25

30

35

**2**

40

45

50

**Intensity (arb. unite)**

For the stoichiometric nominal composition of the CdBa2-x SrxCa2Cu3O8 δ ( x = 0) sample, i.e., without the addition of Sr in the Ba sites, it was found that 67% from CdBa2-x SrxCa2Cu3O8 δ phase (Cd-1223) and 33% fromCdBa2-xSrxCa1Cu2O6+δ (Cd-1212) and some impurity phases of Ca3BaO6 and Ca2CuO3 with vanishingly small concentrations of unknown phases were observed in the XRD pattern. This result indicates that the formation of the Cd-1223 phase is difficult when the sample without any addition. However, on substituting Sr+2 into the Ba+2 sites in the general composition CdBa2-x SrxCa2Cu3O8+δ, followed by the same conditions, a gradual disappearance of the Cd-1212 phase and the formation of the Cd-1223 phase(76% Cd-1223) was observed in the x = 0.20sample. The nearly single phase (86% Cd-1223) of the series CdBa2-x SrxCa2Cu3O8+δwas obtained for the x = 0.30 sample. The lattice parameter values, c/a, volume fraction V2223ph, mass density and shore hardness for different composition of CdBa2-x SrxCa2Cu3O8 δ samples are shown in table-1. It was found from this table that by increasing of the Sr concentrations of all our samples producing changes on the lattice constant, volume fraction (Vphase), c/a parameter, mass density ρm, and shore hardness. It was found from figures (2,3 and 4), the ratio of lattice parameters c/a, the volume fraction (Vphase)and shore hardness are increasing by increasing the Sr concentration, while figure 5 shows that mass density ρm decreasing with increasing the Sr concentration.

Table1. Values of transition temperature Tc(offset),lattice parameter, c/a, volume fraction V2223ph, mass density and shore hardness for the samples for different composition of Bi2-x SbxSr2Ca2Cu3O10+δ

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| *X* | *Tc(offset)( K)* | *a(A0)* | *c(A0)* | *c/a* | V1223ph | *V1212ph* | *Vnknownph* | *Mass density* | *Hardness Shore* |
| 0.0 | 104 | 4.4632 | 14.599 | 3.270 | 67% | 29% | 4% | 6.21 | 48.05 |
| 0.2 | 106 | 4.4758 | 14.688 | 3.281 | 76% | 21% | 3% | 6.05 | 50.87 |
| 0.3 | 113 | 4.4951 | 15.799 | 3.514 | 86% | 11% | 3% | 5.978 | 76.25 |

**C/a**

Fig. 2. Relation of c/a as function ofSr concentration for CdBa2-x SrxCa2Cu3O8 δ with x varying from 0 to 0.3

3.55

3.5

3.45

3.4

3.35

3.3

3.25

3.2

0

0.1

0.2

**Sr Content**

0.3

0.4

100%

80%

60%

40%

20%

0%

0

0.1

0.2

**Sr Content**

0.3

0.4

**V1223ph**

Fig. 3. Relation of volume fraction V(1223)ph as function of Sr concentration for CdBa2-x SrxCa2Cu3O8 δ with x varying from 0 to 0.3

100

80

60

40

20

0

0

0.1

0.2

**Sr Content**

0.3

0.4

**Hardness Shore**

Fig. 4. Variation of Shore hardness with Sr- content for CdBa2-x SrxCa2Cu3O8 δ

Fig. 5. Relation of mass density as function of Sr concentration for CdBa2-x SrxCa2Cu3O8 δ with x varying from 0 to 0.3

6.25

6.2

6.15

6.1

6.05

6

5.95

0

0.1

0.2

**Sr Content**

0.3

0.4

**Mass Density**

The resistivity of all samples vs. temperature has shown a metal like behaviour. Figure 6, illustrated the normalized resistivity vs. temperature deferent Sr concentrations, from which it can be noticed that the increasing of Tc (offset) value against the Sr-doped sample (Sr =0.30) from 106K to 113 K respectively. The critical transition temperature, when (x= 0.0, 0.20, and 0.30)) HTSC phases were found to be 104, 106 and 113 K respectively, see table-1.

The variation in real (ε') and imaginary (ε") parts for the values of dielectric constant as a function of photon energy for CdBa2-x SrxCa2Cu3O8+δ with x varying from 0 to 0.3 are shown in figures (7 and 8), respectively. From those figures we can deduce that the real part of the dielectric constant behaviour nearly similar for all the range of the wavelength spectrum to that of the refractive index because of the real part depends mainly on refractive index according to the equation (ε" = n2-K2).While the imaginary part depend on values of extinction coefficient are related to absorption coefficient variation. Also, we can see the real part values are higher than those of the imaginary part [13].

7

6

5

4

3

2

1

0

X = 0.0

X = 0.20

X = 0.30

80

130

180

**Temperture(K)**

230

280

**Resistivity (Ohm.cm)x10‐6**

Fig. 6. Relation of Resistivity as function of temperature for CdBa2-x SrxCa2Cu3O8 δ with x varying from 0 to 0.3

Fig. 7. Real part of dielectric constant behaviour verses photon energy of CdBa2-x SrxCa2Cu3O8 δ

0

 0.2

0.3

**200**

**150**

**100**

**50**

**0**

**50 1000 100000 300000 500000 700000 100000**

**Frequency(Hz)**

**Dielectric constant(e)**

as a function of Sr content (x=0.0, 0.2 and 0.3)

 0

 0.2

0.3

**145**

**135**

**125**

**115**

**105**

**95**

**85**

**75**

**65**

**55**

**45**

**35**

**25**

**15**

**5**

**-5**

**50 100000 300000 500000 700000 100000**

**Frequency(Hz)**

**Loss factor()**

Fig. 8. Imaginary part of dielectric constant behaviour verses photon energy of CdBa2-x SrxCa2Cu3O8 δ

as a function of Sr content (x=0.0, 0.2 and 0.3)

Scanning electron micrographs (SEM) with secondary electrons are taken to the CdBa2-x SrxCa2Cu3O8 δ with varying x which is shown in Figure 9. It has been discovered that different strontium concentration drives to different sizes grain. It can be also seen from Fig.8 the formations of three phases are characterized by thin elongated grains with orientation that is not preferred and within SEM magnification limits. Specimen of 1223- phase has grains larger than 1212 phase, due to the increase of the contact areas which leads to a larger density, as explained in table 1.



Fig. 9. SEM micrographs as function of Sr concentration for CdBa2-x SrxCa2Cu3O8 δ with x varying from 0 to 0.3

## Conclusion

In this paper, the strontium substitution at the Bi site of CdBa2-x SrxCa2Cu3O8 δ with varying of x effect on the physical properties are presented. The XRD data shows that all samples polycrystalline with tetragonal structure. It has been found that the increasing of the Sr concentrations producing changes on the lattice constant, volume fraction (Vphase), c/a parameter, mass density ρm, and shore hardness. It has been found that the optimum Tc(offset)= 1130 K for CdBa2-x SrxCa2Cu3O8 δ with x = 0.30.It has been discovered that the change of dielectric constant, dielectric factor and tangent loss are produced from the change of the Sr concentration.

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